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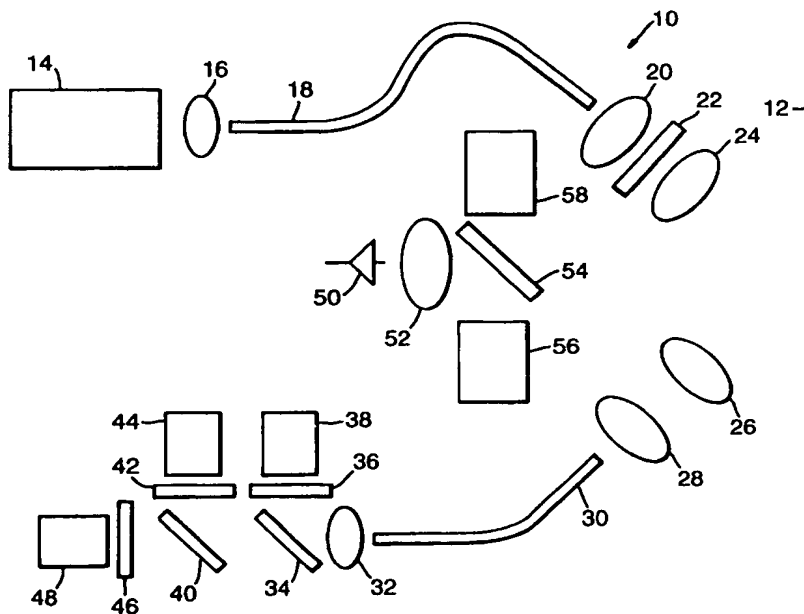
(57) Abstract

The thickness of an organic material on a substrate, such as an oil film on a metal sheet (12), is monitored by illuminating the film with ultraviolet radiation (14) and measuring the fluorescent light intensity (44, 48) from the organic material. The reflectivity of the substrate is also monitored, and used in calculating the thickness. The technique is also applicable where the substrate is moving.

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Monitoring Oil Films

This invention relates to a method and an apparatus
for monitoring the thickness of an organic material, such
5   as an oil film, on a surface of a substrate, for example
on material in a rolling mill, using fluorescence.

In hot metal rolling, whether of ferrous or non-
ferrous metals, lubrication by a rolling oil in the bite
10 of the rollers is believed to play an important role in
the finish attained. An aim of the invention is to
provide a way of monitoring and measuring the thickness
of an oil film on a roller as it leaves the bite. Other
applications are the measurement of oil film thickness
15 applied to steel sheet in preparation for the automotive
industry and the detection of residual oils. A problem
with such measurements is that fluorescence intensities
depend on the characteristics of the surface of the
sheet, such as its reflectivity. This invention provides
20 a scheme for compensating for these quantities.

According to the present invention there is provided
a method of monitoring the thickness of an organic
material on a surface of a substrate, the method
25 comprising illuminating a region of the surface with
radiation of a suitable wavelength to cause fluorescence
in the organic material, detecting the intensity of the
fluorescent radiation, and also measuring the intensity
of radiation reflected from that region of the surface.
30

The reflected radiation may originate from the same
illumination as is used to generate the fluorescence, or
may originate from a different source; the preferred
method also comprised illuminating the surface with a
35 reference beam of a wavelength which is substantially not
absorbed by the organic material, and detecting the

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intensity of the reflected reference beam.

The invention also provides an apparatus for
performing such a method.

5

The source of radiation to cause fluorescence
desirably produces ultraviolet light, and the preferred
source is a pulsed xenon flash lamp. This is desirably
combined with a filter so the surface is only illuminated
10 with suitable u-v wavelengths to cause fluorescence.
Where there is a reference beam, it is preferably
incident substantially normally to the surface.

For detecting the reflected radiation the preferred
15 detector is a photodiode. In a preferred arrangement the
intensities of both the reflected reference beam and the
reflected fluorescence-causing radiation are detected.
To detect the latter a photodiode is desirably combined
with a filter similar to that used in the source.
20

The fluorescent radiation is of much lower intensity
and may be detected by a photomultiplier or an
intensified diode array, desirably combined with a filter
to select only the desired fluorescent wavelengths. This
25 filter might be replaced by a monochromator. If the
radiation source is pulsed, then the fluorescent
radiation detector is desirably gated in synchronisation
with the pulses, so that the effect of any ambient light
is suppressed.
30

The reflectivity of the surface of the substrate
affects the intensity of the fluorescent light in two
ways: a highly reflective surface reflects the incident
radiation back through the organic layer, causing more
35 fluorescence, and also reflects some of the fluorescent
radiation that was initially travelling away from the

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collection optics. Measuring the reflected radiation
enables these effects to be taken into account.

5 The invention will now be further and more
particularly described, by way of example only, and with
reference to the accompany drawings in which:

Figure 1   shows a schematic diagram of an apparatus
            for monitoring oil thickness on a metal
10   sheet;

Figure 2   shows graphically how the intensity of the
            fluorescent light varies with oil film
            thickness, for two different oils;
15

Figure 3   shows graphically how the intensity of the
            fluorescent light varies with the
            intensity of the reflected radiation (for
            oil 2); and
20

Figure 4   shows graphically how the ratio of
            fluorescent light intensities at two
            specified wavelengths varies with
            temperature, for two different oils.
25

Referring to Figure 1, an apparatus 10 for measuring
the thickness of oil on a metal sheet 12 during a rolling
operation includes a xenon flash lamp 14 which operates
at 8 Hz giving flashes of duration about 10 µs. The lamp
30 14 emits radiation in both the visible and ultraviolet
parts of the spectrum. The radiation is focused using
lens 16 into an optical fibre 18 to transmit it to the
measurement region, and is then collimated using a lens
20 and passed through a type UG11 filter 22 to select
35 ultraviolet wavelengths in the range about 290 nm to 370
nm. The filter 22 also removes any fluorescence

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generated in the transmission fibre 18. A lens 24 focuses the u-v radiation onto the sheet 12.

Scattered u-v and fluorescent light are collected by
5 lens 26 and focused into a transmission fibre 30 using a lens 28. Output from the transmission fibre 30 is collected and focused onto three detectors 38, 44 and 48 by a lens 32. Beam splitter 34 deflects onto the detector 38 through a type UG11 transmission filter 36
10 any reflected u-v radiation that has been collected. Beam splitter 40 reflects a portion of the fluorescent light through a filter 42 onto the detector 44, the remainder of the fluorescence passing through a filter 46 onto the detector 48. The two fluorescence signals may
15 be used to calculate oil temperature as well as film thickness. The filters 42 and 46 that are used will depend to a certain extent on the oil, but an example would be a 5 nm bandpass filter at 410 nm for filter 42 and an equivalent one centred at 385 nm for filter 46.
20 The detectors 44 and 48 are gated to operate in synchronism with the 10 µs flashes emitted by the flash lamp 14.

The detector 48 could be a diode array incorporating
25 512 diodes. For some purposes it is preferable to replace the filter 46 with a monochromator (not shown) and remove the beam splitter 40; for example by using a monochromator which gives a dispersion of 70 nm over the diode array 48 the spectrum of the fluorescent light can
30 be observed.

The detectors 44 and 48 are used to measure the intensity of the fluorescent light. This will depend upon the composition of the oil, and in some cases it may
35 be found necessary to add a fluorescent component such as a rhodamine dye. However, rolling oils studied have been

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found to fluoresce without any such addition, possibly
due to the presence of aromatic compounds. For example
oils referred to as oil 1 and oil 2 were both found to
have fluorescent peaks between 385 and 390 nm, and
5 between 400 and 410 nm, though of different intensities.
The intensity and the shape of the fluorescent spectrum
of a particular oil also depend upon its temperature, but
this can be taken into account by a calibration
experiment for the oil in question, determining the
10 temperature dependence of the oil fluorescence intensity,
and measuring the temperature of the oil during
operation. This may be done by monitoring two
appropriate regions of the fluorescent spectrum and
measuring the ratio between the intensities at the two
15 wavelengths. As shown in Figure 4, these ratios can vary
smoothly with temperature; the graph shows the ratio
between 393 nm and 384 nm for oil 1, and the ratios
between 411 nm and 385 nm and between 411 nm and 403 nm
for oil 2.
20

Referring now to Figure 2, the intensity, I , of the
fluorescent light increases with the thickness, ℓ, of the
oil film in the way that can be deduced from Beer-
Lambert's law, such that:
25

$$I = A (1 - e^{-B\ell})$$

where A is an experimental scaling factor, and B depends
on the oil. The values of B for oil 1 and oil 2 were
30 $8.43 \times 10^{-3} \ \mu m^{-1}$ and $9.78 \times 10^{-3} \ \mu m^{-1}$ respectively. These
results are at room temperature, and are determined from
plots such as Figure 2.

When the sheet 12 is moving, the reflectivity cannot
35 be assumed to be constant; and the reflectivity affects

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not only the intensity of the fluorescent light (measured
by the detectors 44 and 48), but also the intensity of
the scattered u-v (measured by detector 38). Referring
now to Figure 3, this shows how the fluorescent light
5   intensity varies with the reflected u-v signal for a 1 μm
thick film of oil 2. Very similar curves have been found
with other oils, and they all fit the equation:

$$I = G(1+3\rho^{z})$$

10

where ρ is the proportion of the u-v radiation which is
reflected, G a scaling factor and z found empirically to
be about 0.3.

15   Hence the intensity of the fluorescence can be
corrected for changes in reflectivity, if the
reflectivity is measured independently. In the apparatus
10 of Figure 1 the reflectivity is monitored by focusing
the light from a red LED 50 using a lens 52 onto the
20   measurement point on the sheet 12. The LED output power
is monitored using a beam splitter 54 and a detector 56,
whilst the light reflected from the sheet 12 is monitored
using a detector 58. The wavelength of the light is such
that it experiences negligible absorption by the oil film
25   on the sheet 12.

Measurement of the reflectivity in this way allows
the reflected u-v radiation measured by detector 38 to be
used to correct for experimental set up factors such as
30   excitation energy and optics collection efficiency. The
way this is done is outlined below.

The technique is based on relating intensities of
fluorescence to oil film thickness and so careful
35   calibration must be carried out. An initial calibration

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signal is collected from a fluorescent marker placed at the measurement position at a known temperature. This marker should be selected to have a fluorescence equivalent to a given oil film thickness; and should be

5 optically thin so that the reflection of light from the substrate surface can be recorded. For example the fluorescence from a 1 mm thick layer of a 1.6 mM solution of pyrene in dodecane is equivalent to that from a film of oil 1 about 4 μm thick. The fluorescent light and

10 reflected radiation from this marker are collected through the same optical system and recorded separately. At the same time, the output and reflected light (from the probed sample position) of the reference beam that is not absorbed by the marker or oil is recorded. By then

15 monitoring these same quantities when taking sample measurements, changes in surface reflectivity, incident u-v intensity, and coupling into the optical system can be accounted for and the fluorescent light can be quantitatively related to oil film thickness. This is

20 outlined in the equations below.

The amount of light transmitted, I_1, through a film of thickness ℓ is given by

$$I_1 = I_0^{-B\ell}$$

25

where I_0 is the initial excitation light intensity, and B is a constant. The amount of light absorbed is given by I_0-I_1, and the fluorescence intensity, I , is proportional to the amount of light absorbed, so that

30                  $$I = KI_0\left(1 - e^{-B\ell}\right)$$

where K accounts for experimental factors such as collection efficiency, etc.

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Hence, as discussed earlier

$$I = A\left(1 - e^{-Bl}\right)$$

where A is an experimental scaling factor, and B depends
on the absorption characteristics of the oil. However,
in this application the film is usually adhering to a
reflective surface which will alter the amount of light
observed, because of reflection of both excitation
(u-v) and fluorescent light. Laboratory experiments have
shown that the fluorescence observed is empirically
modified as follows:-

$$I = A\left(1 - e^{-Bl}\right)\left(1 + 3 \times \left(\frac{R_1}{R_0}\right)^z\right)$$

where R_0 and R_1 are the intensities of the incident and
reflected u-v radiation. The value of z has been found
to be the same (~0.3) for all oils studied, but its
precise value may depend on the collection method of the
reflected light.

Rearranging this equation leads to an expression for
calculating film thickness from fluorescence intensity:

$$l = \frac{-1}{B} Ln\left(1 - \frac{I}{A\left(1 + 3 \times \left(\frac{R_1}{R_0}\right)^z\right)}\right)$$

The experimental factor, A, can be initially determined
by measuring the fluorescence from the standard
calibration solution at a known temperature. The value
of B is that for the oil to be investigated (pre-
determined in laboratory experiments). The reflectivity
of the surface R_1/R_0, is desirably replaced in the above

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equation by S_1/S_0 where S_0 and S_1 are the intensities of
the incident and reflected radiation with the light from
the reference source 50, as this is less likely to be
affected by any changes in optical coupling.

5

The parameter A, ideally a constant, is desirably
replaced by $A_x f_t$ where f_t is a factor to account for the
different temperature the oil is at compared to the
calibration experiment. Its value is calculated from
10 laboratory experiments measuring the oil fluorescence as
a function of temperature. The temperature is determined
using the ratio of two oil fluorescence wavelengths. If
the measurements are at the same temperature as the
calibration, $f_t = 1$. A_x is the new experimental factor
15 that may have changed from the initial calibration
experiment due to variations in excitation energy,
collection efficiency, etc. It is related to the
original factor by

20
$$A_x = A\left(\frac{R_1}{R_1^*}\right)\left(\frac{S_1^*\!\Big/S_0^*}{S_1\!\Big/S_0}\right)$$

where the asterisked values are those measured with the
standard calibration solution. So, after the initial
calibration the system is effectively continuously
25 internally calibrated relative to the initial calibration
experiment, to give accurate film thicknesses.

This method has been shown to be able to measure
thicknesses of less than 0.1 μm, and to be accurate to
30 within 10%.

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Claims

1. A method of monitoring the thickness of an organic
material on a surface of a substrate (12), the method
5 comprising illuminating a region of the surface with
radiation (14, 22) of a suitable wavelength to cause
fluorescence in the organic material, detecting (44, 48)
the intensity of the fluorescent radiation, and also
measuring (38, 50) the intensity of radiation reflected
10 from that region of the surface, and determining the
thickness from the values of the fluorescent intensity
and the reflected intensity.

2. A method as claimed in claim 1 wherein the intensity
15 of reflected radiation originating from the fluorescence-
causing illumination is measured (38).

3. A method as claimed in claim 1 or claim 2 wherein
the region of the substrate is also illuminated with a
20 non-fluorescence-causing reference beam (50), and the
intensity of the reflected reference beam is measured
(58).

4. A method as claimed in claim 3 wherein the reference
25 beam is incident substantially along a normal to the
surface.

5. A method as claimed in any one of the preceding
claims wherein the fluorescence-causing illumination (14)
30 is provided in brief flashes and the means (44, 48)
detecting the fluorescent radiation is gated to operate
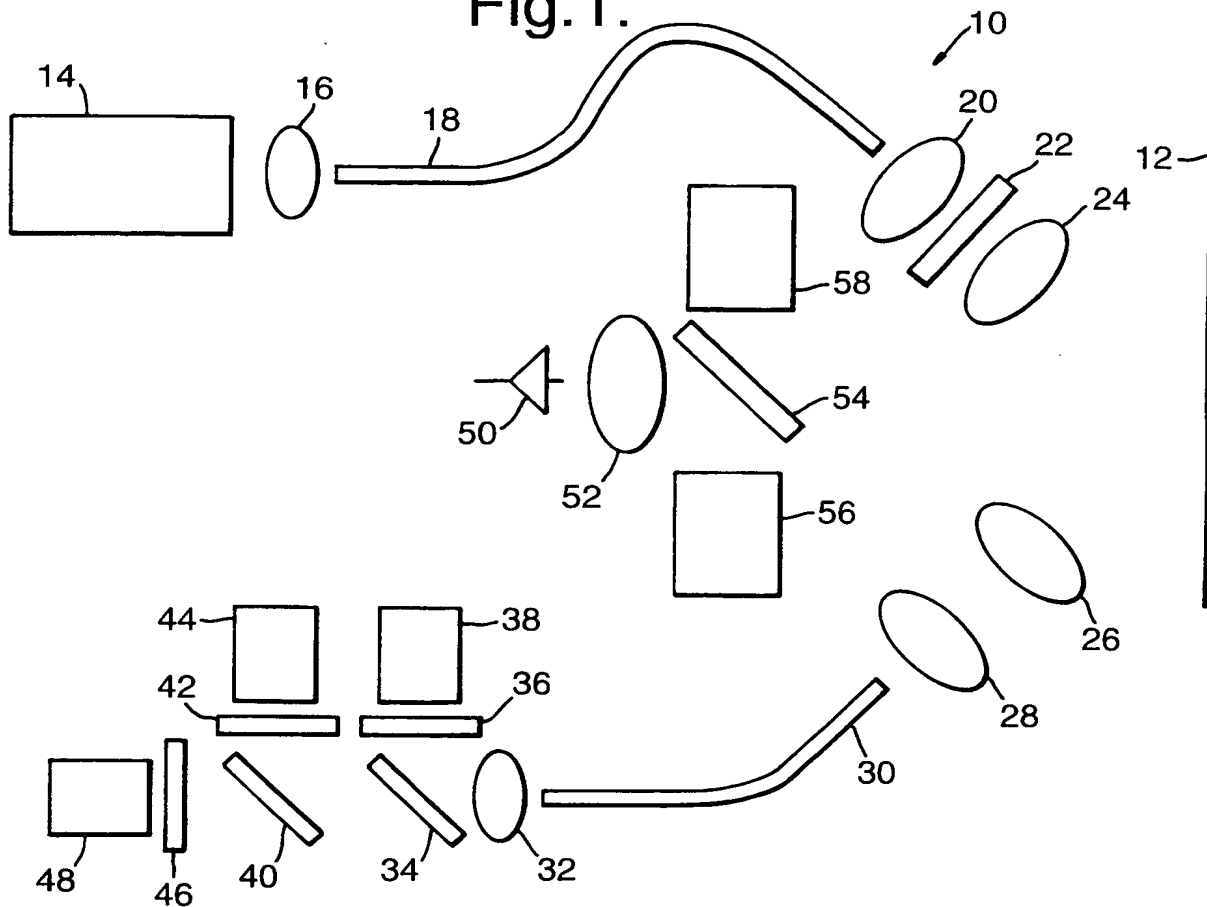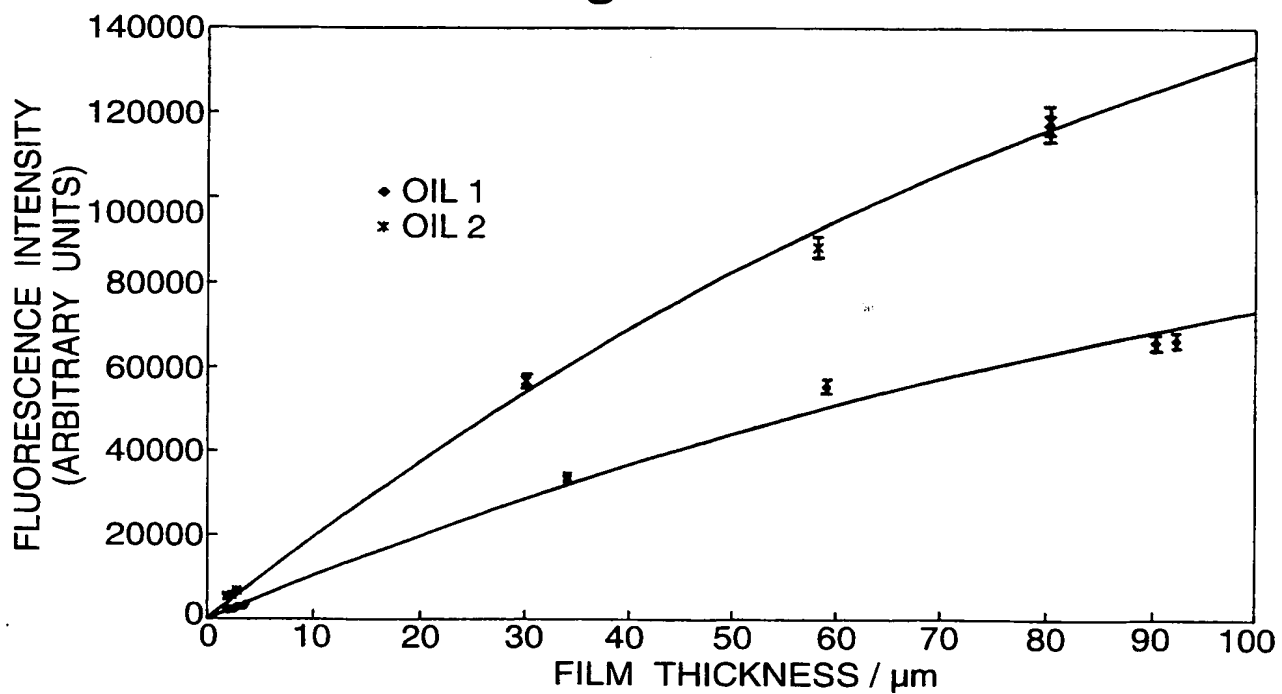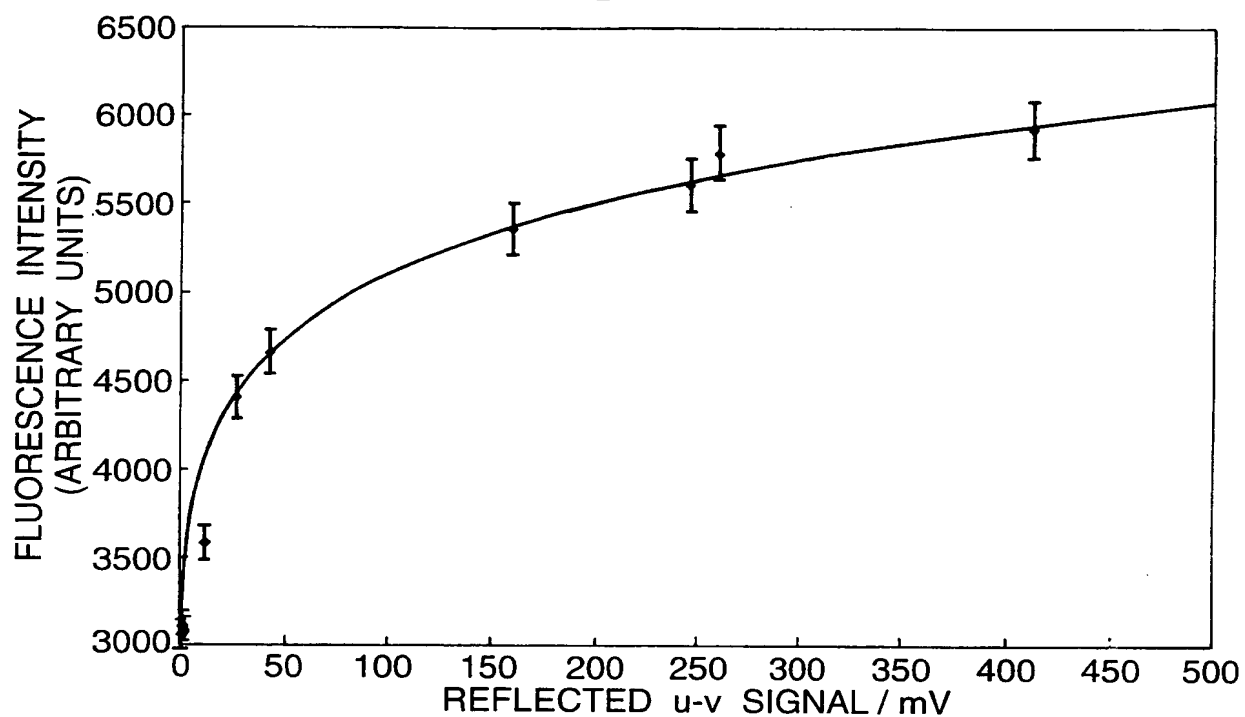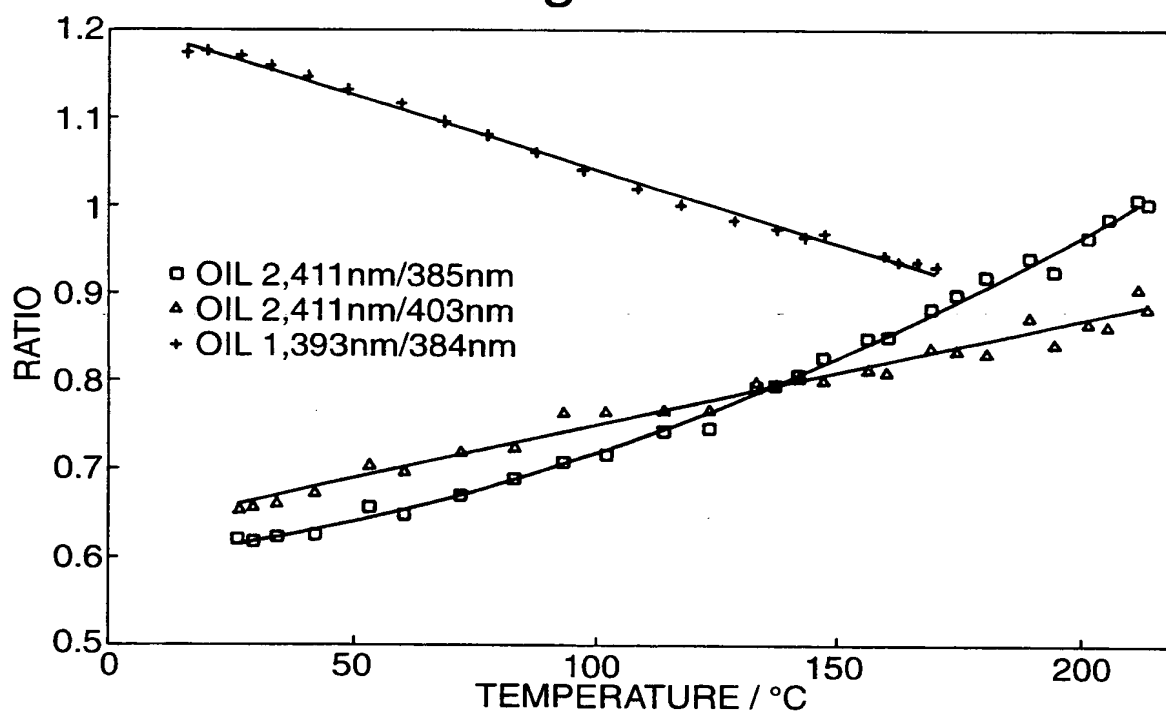in synchronism with those flashes.

Fig.1.

Fig.2.

Fig.3.

Fig.4.

INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6   G01N21/64

According to International Patent Classification(IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6   G01N   G01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 30 38 107 A (BETR FORSCH INST ANGEW FORSCH) 29 April 1982 see claims 1,7,8; figures ---	1,4,5
A	WO 93 22655 A (THIOKOL CORP ;PEARSON LEE H (US); JOHNSON KENDALL B (US); DOYLE TI) 11 November 1993 see claims 15,16 ---	1,5
A	US 4 956 558 A (BATISHKO CHARLES R ET AL) 11 September 1990 see column 1, line 43 – column 2, line 31 see figures -----	1

☐ Further documents are listed in the continuation of box C.   ☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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